Table 2. *Selected geometric parameters* (Å, °)

N1—C2	1.396 (5)	C8—N9	1.342 (5)
N1—C6	1.405 (4)	C8—C3′	1.486 (5)
N1—C1*x*	1.480 (5)	C1′—C2′	1.519 (7)
C2—N3	1.378 (5)	C1′—C8′	1.562 (7)
C2—O2*x*	1.217 (4)	C1′—C9′	1.523 (5)
N3—C4	1.379 (4)	C2′—C3′	1.560 (5)
N3—C3*x*	1.464 (6)	C3′—C4′	1.559 (5)
C4—C5	1.358 (5)	C3′—C7′	1.567 (6)
C4—N9	1.361 (5)	C4′—C5′	1.530 (7)
C5—C6	1.419 (5)	C5′—C6′	1.567 (8)
C5—N7	1.388 (4)	C5′—C9′	1.522 (6)
C6—O6*x*	1.220 (5)	C6′—C7′	1.529 (5)
N7—C8	1.351 (5)	C7′—C8′	1.526 (6)
C2—N1—C6	126.8 (3)	C4—C5—C6	123.3 (3)
C2—N1—C1*x*	115.8 (3)	C4—C5—N7	104.9 (3)
C6—N1—C1*x*	117.3 (3)	N1—C6—C5	111.7 (3)
N1—C2—N3	117.0 (3)	N1—C6—O6*x*	120.7 (3)
N1—C2—O2*x*	121.6 (4)	C5—C6—O6*x*	127.6 (3)
N3—C2—O2*x*	121.4 (4)	C5—N7—C8	106.3 (3)
C2—N3—C4	119.4 (3)	N7—C8—N9	112.9 (3)
C2—N3—C3*x*	119.7 (3)	N7—C8—C3′	122.3 (3)
C4—N3—C3*x*	120.9 (3)	N9—C8—C3′	124.8 (3)
N3—C4—C5	121.8 (3)	C4—N9—C8	103.0 (3)
C5—C4—N9	112.8 (3)		
N7—C8—C3′—C2′	58.3 (4)	N7—C8—C3′—C7′	178.5 (3)
N7—C8—C3′—C4′	−58.4 (4)	N9—C8—C3′—C7′	0.3 (5)

The atomic coordinates of H atoms were geometrically recalculated after each refinement cycle and included in the structure-factor calculations. The B_{iso} of each H atom was reset to 1.30 × $B_{eq}(X)$ after each refinement cycle, where $B_{eq}(X)$ is B_{eq} of the atom to which the H atom is connected.

Data collection: *CAD*-4 *SDP-Plus* (B. A. Frenz & Associates, Inc., 1985). Cell refinement: *CAD*-4 *SDP-Plus*. Data reduction: *CAD*-4 *SDP-Plus*. Structure solution: *MULTAN*11/82 (Main *et al.*, 1982). Structure refinement: *CAD*-4 *SDP-Plus*. Molecular graphics: *ORTEP*II (Johnson, 1976). Preparation of material for publication: *CAD*-4 *SDP-Plus*.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AS1162). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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On the Structure of (13-Methyl-1,4,7,8,13,-13b-hexahydro[1′,2′]oxazepino[2′,3′:1,2]-pyrido[3,4-*b*]indol-1-yl)methanol

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Abstract

The crystal structure of $C_{17}H_{20}N_2O_2$, originally described in space group *Pc* [Ohishi, Sakamoto, Harusawa, Yoneda & Kurihara (1995). *Acta Cryst.* C**51**, 135–139], is better described and refined in $P2_1/c$. Revised coordinates and bond lengths are given. Whereas it was originally reported that the structure is based on four independent molecules in the asymmetric unit, three of one chirality and one of the opposite chirality, both the original space group (*Pc*) and the revised space group ($P2_1/c$) are enantiomorphic and the structure must contain an equal number of both configurations.

Comment

My attention – and, I suspect, that of many other readers – was first drawn to this article (Ohishi, Sakamoto, Harusawa, Yoneda & Kurihara, 1995; hereafter, OSHYK) by the authors' claim that 'four molecules exist as an enantiomeric mixture with a ratio of 3:1 in a single crystal' whereas the reported space group, *Pc*, is achiral and hence the structure must contain equal numbers of both enantiomers. Further examination suggested that the structure might be better described in space group $P2_1/c$, with but two molecules in the asymmetric unit; the coordinates reported by OSHYK indicate that corresponding atoms in the molecules *A* and *B*, and in molecules *C* and *D*, are related to one another by an approximate center of symmetry at (0.485, 0.250, 0.492). Another strong indication that the structure was described incorrectly as non-centrosymmetric lies in the reported bond lengths (OSHYK, Table 2); corresponding bonds in the four molecules differ by as much as 0.22 Å in the face of e.s.d.'s of 0.01 Å or less.

Refinement in $P2_1/c$ was based on the 4021 F_o values in the supplementary material (OH1042). Included were two reflections, 010 and 050, which should be extinguished in $P2_1/c$; both had F_o and F_c values among the smallest in the list. H atoms were first placed in assumed positions, and then included in the refinement. Convergence was reached quickly at R = 0.0629 for

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540 parameters, surely sufficiently close to the value of 0.0615 reported by OSHYK for 1078 parameters in *Pc*. Final coordinates are listed in Table 1.

Bond lengths for the two molecules in the asymmetric unit are given in Table 2, together with the range of bond lengths reported by OSHYK for the four independent molecules in their *Pc* refinement. The comparison is striking; whereas corresponding bonds differed by as much as 0.22 Å (or $\sim 20\sigma$) in the earlier description, no difference in the new description is as great as 3σ. We have here another striking example of the well known result that refinement of a centrosymmetric structure in a non-centrosymmetric space group, even if apparently successful, may lead to severe distortions because of the inherent near-singularities in the situation.

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
C(1*A*)	0.4590 (2)	0.0010 (5)	−0.1308 (1)	0.0384 (7)
C(2*A*)	0.5029 (2)	−0.1516 (5)	−0.0906 (1)	0.0475 (8)
C(3*A*)	0.4836 (2)	−0.2464 (5)	−0.0512 (1)	0.0549 (9)
C(4*A*)	0.4090 (2)	−0.2476 (7)	−0.0392 (1)	0.0614 (10)
O(5*A*)	0.3552 (1)	−0.1320 (4)	−0.0763 (1)	0.0546 (5)
N(6*A*)	0.3517 (1)	−0.2316 (4)	−0.1265 (1)	0.0437 (6)
C(7*A*)	0.2735 (2)	−0.2889 (6)	−0.1477 (1)	0.0573 (9)
C(8*A*)	0.2703 (2)	−0.4235 (6)	−0.1957 (1)	0.0552 (9)
C(9*A*)	0.3165 (2)	−0.3188 (5)	−0.2286 (1)	0.0438 (7)
C(10*A*)	0.3176 (2)	−0.3601 (5)	−0.2809 (1)	0.0448 (7)
C(11*A*)	0.2834 (2)	−0.5120 (6)	−0.3177 (1)	0.0564 (9)
C(12*A*)	0.2967 (2)	−0.5042 (7)	−0.3667 (2)	0.0664 (10)
C(13*A*)	0.3445 (2)	−0.3492 (7)	−0.3800 (1)	0.0646 (10)
C(14*A*)	0.3793 (2)	−0.1989 (6)	−0.3442 (1)	0.0547 (9)
C(15*A*)	0.3662 (2)	−0.2068 (5)	−0.2946 (1)	0.0433 (7)
N(16*A*)	0.3933 (1)	−0.0796 (4)	−0.2518 (1)	0.0401 (6)
C(17*A*)	0.3632 (1)	−0.1522 (5)	−0.2115 (1)	0.0385 (7)
C(18*A*)	0.3780 (2)	−0.0634 (5)	−0.1575 (1)	0.0397 (7)
C(19*A*)	0.4367 (2)	0.1157 (6)	−0.2549 (1)	0.0528 (9)
C(20*A*)	0.4607 (2)	0.2254 (5)	−0.1063 (1)	0.0456 (8)
O(21*A*)	0.4367 (1)	0.3765 (4)	−0.1473 (1)	0.0538 (6)
C(1*C*)	0.0551 (2)	0.2432 (5)	−0.3970 (1)	0.0444 (7)
C(2*C*)	0.1225 (2)	0.0931 (5)	−0.3892 (2)	0.0535 (9)
C(3*C*)	0.1599 (2)	−0.0018 (6)	−0.3470 (2)	0.0646 (11)
C(4*C*)	0.1434 (2)	−0.0068 (7)	−0.2953 (2)	0.0752 (12)
O(5*C*)	0.0768 (1)	0.1092 (4)	−0.2929 (1)	0.0637 (6)
N(6*C*)	0.0171 (1)	0.0106 (4)	−0.3311 (1)	0.0500 (6)
C(7*C*)	−0.0406 (2)	−0.0519 (7)	−0.3032 (1)	0.0626 (10)
C(8*C*)	−0.0976 (2)	−0.1864 (6)	−0.3402 (1)	0.0569 (9)
C(9*C*)	−0.1179 (2)	−0.0765 (5)	−0.3912 (1)	0.0456 (7)
C(10*C*)	−0.1800 (2)	−0.1107 (5)	−0.4334 (1)	0.0460 (7)
C(11*C*)	−0.2387 (2)	−0.2589 (6)	−0.4434 (2)	0.0602 (9)
C(12*C*)	−0.2911 (2)	−0.2426 (7)	−0.4890 (2)	0.0684 (10)
C(13*C*)	−0.2880 (2)	−0.0826 (7)	−0.5253 (2)	0.0631 (10)
C(14*C*)	−0.2306 (2)	0.0660 (6)	−0.5165 (1)	0.0550 (9)
C(15*C*)	−0.1775 (2)	0.0480 (5)	−0.4706 (1)	0.0454 (7)
N(16*C*)	−0.1149 (1)	0.1739 (4)	−0.4523 (1)	0.0471 (6)
C(17*C*)	−0.0790 (2)	0.0946 (5)	−0.4041 (1)	0.0420 (7)
C(18*C*)	−0.0083 (2)	0.1803 (5)	−0.3698 (1)	0.0435 (7)
C(19*C*)	−0.0996 (2)	0.3698 (6)	−0.4789 (2)	0.0598 (10)
C(20*C*)	0.0850 (2)	0.4686 (5)	−0.3800 (2)	0.0539 (9)
O(21*C*)	0.0269 (1)	0.6204 (4)	−0.3984 (1)	0.0658 (7)

Table 2. *Bond lengths* (Å) *for this refinement, and the range of values reported for the earlier Pc refinement*

	Molecule		*Pc* structure*
	A	*C*	Range
C(1)—C(2)	1.516 (4)	1.532 (5)	1.473–1.569
C(1)—C(18)	1.556 (4)	1.560 (4)	1.503–1.608
C(1)—C(20)	1.536 (4)	1.535 (5)	1.496–1.559
C(2)—C(3)	1.316 (5)	1.313 (5)	1.252–1.419
C(3)—C(4)	1.484 (5)	1.469 (6)	1.398–1.545
C(4)—O(5)	1.423 (4)	1.440 (5)	1.312–1.525
O(5)—N(6)	1.452 (3)	1.450 (3)	1.450–1.475
N(6)—C(7)	1.472 (4)	1.481 (5)	1.438–1.518
N(6)—C(18)	1.478 (4)	1.471 (4)	1.455–1.497
C(7)—C(8)	1.509 (5)	1.517 (5)	1.441–1.588
C(8)—C(9)	1.500 (5)	1.483 (5)	1.468–1.519
C(9)—C(10)	1.413 (4)	1.423 (4)	1.384–1.459
C(9)—C(17)	1.359 (4)	1.370 (4)	1.340–1.407
C(10)—C(11)	1.399 (5)	1.403 (5)	1.352–1.467
C(10)—C(15)	1.413 (4)	1.402 (4)	1.335–1.494
C(11)—C(12)	1.372 (5)	1.369 (5)	1.315–1.458
C(12)—C(13)	1.405 (5)	1.392 (6)	1.293–1.513
C(13)—C(14)	1.381 (5)	1.387 (5)	1.393–1.436
C(14)—C(15)	1.388 (4)	1.382 (4)	1.353–1.428
C(15)—N(16)	1.378 (4)	1.390 (4)	1.323–1.438
N(16)—C(17)	1.388 (3)	1.387 (4)	1.343–1.433
N(16)—C(19)	1.467 (4)	1.465 (5)	1.443–1.492
C(17)—C(18)	1.497 (4)	1.509 (4)	1.441–1.571
C(20)—O(21)	1.426 (4)	1.429 (4)	1.374–1.493

* Ohishi *et al.* (1995), Table 2. The reported e.s.d.'s were in the range 0.006–0.01 Å.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and isotropic *B*'s have been deposited with the IUCr (Reference: CR1197). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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